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REPORT SECURITY CLASSIFICATION: UNCLASSIFIED

1b. RESTRICTIVE MARKINGS

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3. DISTRIBUTION/AVAILABILITY OF REPORT

AD-A229 082

5. MONITORING ORGANIZATION REPORT NUMBER(S)

AFOSR-TR- 90 1149

DTIC ELECTE NOV 21 1990 S E D

NAME OF PERFORMING ORGANIZATION: niversity of Minnesota

6b. OFFICE SYMBOL (If applicable)

7a. NAME OF MONITORING ORGANIZATION: AFOSR

ADDRESS (City, State and ZIP Code)
919 University Avenue
St. Paul, MN 55104

7b. ADDRESS (City, State and ZIP Code)
Building 410
Bolling AFB, Washington, DC 20332

NAME OF FUNDING/SPONSORING ORGANIZATION: AFOSR/NE

8b. OFFICE SYMBOL (If applicable): AFOSR/NE

9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER: AFOSR-87-0372

ADDRESS (City, State and ZIP Code)
Building 410
Bolling AFB
Washington, DC 20332-6448

10 SOURCE OF FUNDING NOS.

PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT NO.
61102F	2306	C1	

TITLE (Include Security Classification): High Temperature Superconducting Compound

PERSONAL AUTHOR(S): A. M. Goldman, M. L. Mecartney

TYPE OF REPORT: Final Technical Report

13b. TIME COVERED FROM 9/1/87 TO 9/30/90

14. DATE OF REPORT (Yr., Mo., Day): 10/1/90

15. PAGE COUNT: 12

SUPPLEMENTARY NOTATION

COSATI CODES		
FIELD	GROUP	SUB. GR.

18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
Superconductivity,
Materials
Thin Films, (JS)

ABSTRACT (Continue on reverse if necessary and identify by block number)

High-T_c superconductors have been investigated in both bulk and thin film form. A technique for the *in-situ* preparation of high-T_c superconducting films involving the use of ozone-assisted Molecular Beam Epitaxy has been developed. The procedures seem to be generalizable to the extent that high quality trilayer and multilayer structures which would be useful scientifically and technologically are possible. In addition to the process working with the usual substrates, $SrTiO_3$, YSZ, MgO, and $LaAlO_3$, it has been possible to deposit films on Si substrates without any buffer layer. A bolometer has been successfully fabricated on a thermally isolated SiN substrate coated with YSZ. Very thin and transparent films with relatively high transition temperatures have been prepared. The magnetic properties of bulk polycrystalline and single crystal high temperature superconductors have been measured and reveal important features of flux pinning and anisotropy in these materials. A low temperature scanning tunneling microscope for the investigation of the surfaces of high-T_c superconductors has been developed. Spherical target sputtering as a means of growing high-T_c films has been explored. Studies of electrical transport above and in the vicinity of the transition have revealed features of the fluctuations in the normal state and of the Kosterlitz-Thouless character of the transition.

DISTRIBUTION/AVAILABILITY OF ABSTRACT
UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT. ☐ DTIC USERS ☐

21. ABSTRACT SECURITY CLASSIFICATION: UNCLASSIFIED

NAME OF RESPONSIBLE INDIVIDUAL: Dr. Harold Weinstock

22b. TELEPHONE NUMBER (Include Area Code): 202-767-4933

22c. OFFICE SYMBOL: AFOSR/NE

FORM 1473, 83 APR EDITION OF 1 JAN 73 IS OBSOLETE.

SECURITY CLASSIFICATION OF THIS PAGE

Final Report

HIGH TEMPERATURE SUPERCONDUCTING COMPOUNDS

AFOSR GRANT NO. 87-0372

Accession For	
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Unannounced	☐
Justification______	
By______	
Distribution/	
Availability Codes	
Dist	Avail and/or Special
A-1	

1 October 1990

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TABLE OF CONTENTS

SUMMARY

The major accomplishment of this grant has been to develop a technique for the *in-situ* preparation of high-T_c superconducting films involving the use of ozone-assisted Molecular Beam Epitaxy. The method is highly reliable and reproducible. The procedures seem to be generalizable to the extent that high quality trilayer and multilayer structures which would be useful scientifically and technologically are possible. In addition to the process working with the usual substrates, $SrTiO_3$, YSZ, MgO, and $LaAlO_3$, it has been possible to deposit films on Si substrates without any buffer layer. A bolometer has been successfully fabricated on a thermally isolated SiN substrate coated with YSZ. The latter effort was carried out in collaboration with Honeywell Inc. The deposition process also facilitates the fabrication of very thin and transparent films with relatively high transition temperatures. This has created new opportunities for the study of superconductor-insulator transitions and the investigation of photo-doping with carriers of high temperature superconductors.

A second important accomplishment has involved the measurement of the magnetic properties of bulk polycrystalline and single crystal high temperature superconductors. These investigations have revealed important features of flux pinning and anisotropy in these materials. Other major areas of effort have involved the development of a low temperature scanning tunneling microscope for the investigation of the surfaces of high-T_c superconductors, the development of spherical target sputtering as a means of growing high-T_c films, and the study of electrical transport above and in the vicinity of the transition which has revealed features of the fluctuations in the normal state and of the Kosterlitz-Thouless character of the transition.

I. INTRODUCTION

This report describes the results of research during the period 1 September 1987 to 30 September 1990 conducted under AFOSR Grant No. 87-0372. The work is concerned with high-T_c superconducting compounds. These materials first attracted world-wide attention more than three years ago. The efforts supported under the grant are directed at both the bulk and the thin-film forms of these oxides with the ultimate objective of elucidating the underlying mechanism(s) for the superconductivity of these materials as well as developing processes and structures of technological significance.

The new high-T_c materials, and the prospect of future discoveries involving ultra-high transition temperature superconductivity have greatly broadened interest in superconductivity in both the engineering and scientific communities. Superconducting materials may be offered as a solution to a variety of problems in areas of technology hitherto abandoned to more conventional materials. The prospect of practical superconductivity at liquid nitrogen temperatures (and perhaps higher) could have an enormous impact on a variety of industrial and military technologies in areas relating to energy, electronics and information processing.

It is clear that future technological success is related to the ability to control the chemical composition and morphology and perhaps with the development of the capability to fabricate thin films of prescribed morphology and chemical composition. Although most of the advances in the limiting values of superconducting parameters have come from empirical discoveries associated with highly Edisonian fabrication efforts, optimization of the properties may result from theoretical modeling which should point the way towards future improvements in such properties. The efforts involved in preparing samples of sufficient quality to test theoretical models and answer other scientific questions are closely related to those efforts needed for the development of materials adequate for superconducting technology.

The history of the past three years of research on high temperature superconductors demonstrates that a major on-going technical challenge in the field, in addition to finding additional materials with even higher T_c's, is the preparation of materials of exceptionally high quality. This would permit intrinsic superconducting properties to be determined with a certain degree of confidence, thus facilitating the elucidation of the mechanism for high temperature superconductivity.

The complementary skills and backgrounds of the two principal investigators have been applied to solve both fabrication and analytical problems in order to meet the above challenge. Materials have been fabricated in both bulk and thin film form and characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), and scanning electron microscopy (SEM). These studies have been correlated with investigations of macroscopic superconducting properties such as critical temperatures, transition widths, critical magnetic fields, the Meissner effect, and penetration depths. Progress in the fabrication of thin films has progressed to the point where the fabrication of planar tunneling junctions and proximity effect structures may actually be feasible in the short term.

II. SCIENTIFIC RESULTS

A. Preparation of High-T_c Films by Ozone-assisted Molecular Beam Epitaxy and Related Efforts.

This effort grew out of a program to develop techniques for the growth of high-T_c superconducting films by co-evaporation. The early efforts resulted in the synthesis of $Y_2Ba_4Cu_8O_{20-x}$ films which were post-annealed (Berkley, *et al.*, 1988a). It was then realized that $YBa_2Cu_3O_{7-x}$ films could be prepared *in-situ* without a post-anneal by using an ozone vapor jet as an oxygen source (Berkley, *et al.*, 1988b). In both of these initial works a combination of electron beam and Knudsen vapor sources was employed for the various metallic constituents. It is clear that the use of a well-characterized oxidizing gas is very useful for the fabrication of high-T_c superconducting films. The details of these procedures were described in a comprehensive article published in the *Review of Scientific Instruments* (Berkley, *et al.*, 1989a).

A major improvement on the process followed with the abandonment of electron beam sources for a configuration in which the elemental constituents were deposited entirely using Knudsen Cells. Films of $DyBa_2Cu_3O_{7-x}$ with transition temperatures as high as 89K and with nominal thicknesses down to 35Å were grown *in-situ* using molecular beam epitaxy employing ozone (Johnson, *et al.*, 1990). The growth process, which was carried out at a substrate temperature of 700°C, was successful with a variety of substrates including $SrTiO_3$(100), $SrTiO_3$(110), $LaAlO_3$ (100), MgO(100), and yttria stabilized zirconia (YSZ). The surfaces of these films could be imaged with a scanning tunneling microscope operating at 4.2K, indicating a conducting surface even at low temperatures. Recently we have been able to grow films at a substrate temperature of 600°C directly onto Si substrates *without any preconditioning*. The best film to date prepared in this manner has an onset temperature of 90K, and a zero resistance temperature of 70K. This result implies that ozone-assisted molecular beam epitaxy may be very valuable for the fabrication of hybrid semiconductor-superconductor devices.

A particularly interesting application of the above techniques was the fabrication of a high temperature superconducting microbolometer employing a DyBaCuO film deposited upon a silicon microstructure. This device was found to have a responsivity of 800V/W at 89K and a response time of 1 ms (Stratton, *et al,* 1990).

B. Tunneling Junctions with High Temperature Superconductors.

Work was begun towards the development of tunneling junctions and other heterostructures based on DyBaCuO. First it was discovered that superconducting DyBaCuO could be grown epitaxially on a buffer layer of Dy_2O_3. The latter was prepared in the vacuum chamber by simply closing the shutters on the Ba and Cu sources. Cross-sectional TEM revealed very sharp boundaries between the DyBaCuO and the underlying buffer layer. Then DyBaCuO-Dy_2O_3-DyBaCuO sandwiches were produced and examined by cross-sectional TEM. Again very sharp boundaries were formed, and the structure appeared as a monolithic epitaxial layer when examined using various microstructural characterization techniques. These results are extraordinarily encouraging for the production of tunneling junctions and heterostructures made from high temperature superconductors. The next step will be to implement patterning and produce sandwiches with thin barriers, which should permit tunneling studies to be carried out. This work will be presented at the 1990 Applied Superconductivity Conference and will appear in the Proceedings of that meeting (Beauchamp, *et al.*, 1991).

C. Hemispherical Target Sputtering.

In collaboration with G. Wehner of the Electrical Engineering Department, high quality stoichiometric films of $YBa_2Cu_3O_{7-x}$ were also grown using a unique sputtering technique involving the use of a stoichiometric target of hemispherical shape (Wehner, *et al.*, 1988). This was done in a Hg vapor triode plasma. The method permits the fabrication of stoichiometric films from stoichiometric targets. This process has the advantage that simple modifications will permit significant increases in sputtering rates and the process can be scaled up in size easily. The use of a spherical target in effect permits an averaging over the angular distribution of the sputtering yield of the constituents. The method was also extended to include spherical targets (Wehner, *et al.*, 1989).

D. Magnetic Studies of Bulk High-T_c Superconducting Materials.

These investigations were carried out using a Quantum Design Superconducting SQUID Susceptometer. The first work involved the investigation of the time dependence of the magnetization of polycrystalline samples of YBCO (Tuominen, Goldman and Mecartney, 1988a). A comparison of field-cooled and zero field-cooled magnetizations revealed the existence of a reversibility line. A logarithmic dependence of the decay of the field-cooled magnetization was observed. The temperature dependence of the fractional change of the magnetization was observed to have a peak. This first work was interpreted in the context of a superconducting glass model. It is clear at this time that a critical state model would also work. It was later found that similar results could be obtained from the study of single-crystals samples of $YBa_2Cu_3O_{7-x}$ (Tuominen, Goldman and Mecartney, 1988b).

Research on magnetic properties then turned to the question of magnetic anisotropy. The anisotropies of $Bi_2Sr_2CaCu_2O_y$ and $YBa_2Cu_3O_{7-x}$ were investigated by simultaneously measuring the longitudinal (M_L) and transverse (M_T) components of the equilibrium magnetization of crystals oriented at arbitrary angles with respect to the applied field direction (Tuominen, *et al.*, 1990a). The variations of ML and M_T as a function of orientation, field and temperature were measured. In the regime in which the simple three-dimensional anisotropic London theory is valid it was shown that the ratio of the two magnetizations yields the anisotropic effective mass ration m_3/m_1 directly. This number was found to be 30 and 280 for crystals of $YBa_2Cu_3O_{7-x}$ and $Bi_2Sr_2CaCu_2O_y$ respectively.

The same apparatus was then used to study the relaxation of the longitudinal and transverse components of the remanent magnetic moment of single crystal $Bi_2Sr_2CaCu_2O_y$ as a function of angle (Tuominen, *et al.*, 1990b). When the angle between the applied field and the c-axis is not too large, the remanent moment aligns quickly with the c-axis. At extremely high angles the alignment is slow and at low temperatures the transverse magnetization even increases with time. The temperature dependence of the normalized relaxation rate exhibits two peaks, suggesting the existence of two separate pinning mechanisms.

E. Electrical Transport in TlBaCaCuO Thin Films.

This work was carried out in collaboration with a former student, J. Kang who at the time was a postdoc at Argonne National Laboratory, and is now a staff member at Westinghouse. The first studies involved measurements of in-plane fluctuation-enhanced conductivity of c-axis oriented TlBaCaCuO thin films performed over the temperature range

from T_c to 240K (Kim, *et al.*, 1989a). The results were consistent with two-dimensional fluctuation theory and with a linear dependence of the normal state resistivity on temperature down to $(T-T_c)/T_c$ the order of 0.03. A crossover to three-dimensional fluctuations close to T_c was not found. The width of the superconducting transition appeared to be a measure of a distance over which layers fluctuate in a correlated manner.

The transport properties of the same type of films were then examined within the context of the Kosterlitz-Thouless-Berezinskii model (Kim, *et al.*,1989b). The nonlinear current versus voltage and resistivity versus magnetic field characteristics below the transition temperature together with the exponential inverse-square-root temperature dependence of the resistivity just above T_c were consistent with each other and with the theory. The parametrization of the resistivity data using the Aslamazov-Larkin theory well above the KTB transition was consistent with that of the KTB theory in the transition region.

F. Other Work

In the early days of high-T_c superconductivity, X-ray photoelectron spectroscopy studies were performed on bulk polycrystalline samples of YBCO as a function of temperature through the superconducting transition (Kim, *et al.*, 1988). With decreasing temperature, clear changes were observed in the photoelectron spectrum which were at the time interpreted as signatures of the $Cu^{3+}$and perhaps the Cu^{1+} oxidation states, in addition to the usual Cu^{2+} state. These changes were not observed in samples which had been rendered nonsuperconducting by baking in vacuum.

The fabrication of superconductors using sol-gel synthesis was also investigated (Accibal, *et al*, 1989). A comparison of the use of three different coordination compounds of copper as precursors for the sol-gel synthesis of $YBa_2Cu_3O_{7-\delta}$ has been made. For yttrium, the tris(isopropoxide) was used exclusively, while the use of both Ba(O-*i*-Pr)$_2$ and $Ba(OCH_2Ch_2OEt)_2$ (prepared *in-situ* from Ba metal) as sources for Ba were studied. After dissolving Y(O-*i*-Pr)$_3$, a Ba source, and the copper(I) alkoxide, [Cu(O-*t*-Bu)]$_4$, hydrolysis led immediately to an orange gelatinous solid which yielded $YBa_2Cu_3O_{7-\delta}$ upon firing in oxygen. Copper(II) acetate was found to give heterogeneous mixtures under our conditions and was not further studied. Copper(II) acac (acac = acetylacetonate) yielded the best results. Partially hydrolyzed solutions of $Cu(acac)_2$, $Ba(OCH_2CH_2OEt)_2$, and Y(O-*i*-Pr)$_3$ were spin coated on $SrTiO_3$ (100) and fired under oxygen to give oriented (*b* axis normal to the surface) thin films of $YBa_2Cu_3OO_{7-\delta}$. The onset of superconductivity for the films was 92 K, but they did not reach zero resistance until much lower temperatures.

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V. DISSERTATIONS

A. Doctoral

Kim, D. H., "Transport Properties of High Temperature Superconductor TlBaCaCuO Thin Films," August 1989
Present Employer: Argonne National Laboratory (K. Gray)

Berkley, D. D., "*In-situ* Preparation of Y-Ba-Cu-O Superconducting Thin Films Using Pure Ozone Vapor Oxidation," December 1989
Present Employer: Naval Research Laboratory (S. Wolf)

Tuominen, M., "Anisotropic and Time-Dependent Magnetization of the High Transition Temperature Superconducting Cuprates," July 1990
Present Employer: Harvard University (M. Tinkham)

B. Masters

Hassler, B. A., "High T_c Superconducting Thin Films by Sol-Gel Coating," December, 1988.
Present Employer: Medtronics, Inc.